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# QUARTERLY LETTER REPORT

## Thermochemistry of Selected Compounds
Nonr-3608(00)

by

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## MAJOR ACCOMPLISHMENTS

### A. Aluminum Borohydride Studies

It appeared from some of our preliminary studies that the hydrolysis of aluminum borohydride with water vapor at 0°C or lower minimized the production of the undesirable side product, pentaborane. We, therefore, chose to concentrate our studies at 0°C or slightly higher for convenience. We found it experimentally convenient to operate the calorimeter at 2°C. Modifications were necessary to allow the calorimeter to operate at this temperature in the preferred adiabatic method. The inner bucket containing the reaction vessel and two liters of water also at 2°C was allowed to equilibrate with the jacket to a desired heat leak level. The $\Delta T$ between the jacket and the reaction vessel and environment was kept constant by automatic control. As the reaction was initiated resulting in an increased temperature of the reaction vessel and surrounding water, the jacket water was automatically heated by an immersion heater activated by the relay mechanism of the automatic control. The results exhibited large variations,and the uncertainty was much higher than desired. It appeared that the jacket temperature was fluctuating because of temperature variations of a precooling bath in the experimental setup. Although these fluctuations were small, the high sensitivity of the calorimeter was affected by these fluctuations. We, therefore, sought to eliminate the problem by attempting to operate the calorimeter isothermally. We remodeled the thermistorized control bridge such that the

jacket water would be controlled at one temperature. To check out the method we performed simulated runs by calibrating electrically and by also performing a heat of reaction of magnesium with hydrochloric acid for a chemical check of the electrical method of calibration. From these results, the method appeared promising.

Nine runs of the hydrolysis of aluminum borohydride with water vapor at approximately 2°C were performed. The resultant heat was -227 kcal/mole. The uncertainty of ± 7 kcal/mole is undesirable and too high, and once again we are seeking the reasons for this. We have noted in the preparation of our samples that the distillation and fractionation of the aluminum borohydride appears to be a critical step and may be the cause of our scatter. Our immediate efforts will be concerned with the sample preparation method; in particular, the temperatures employed in the fractionation process along with analytical studies of the products resulting from the hydrolysis of the samples prepared. We will, of course, continue with the heat measurement of these same samples. Calculating the heat of formation of aluminum borohydride using the value of -227 kcal/mole for the heat of reaction plus the known values of the heats of formation of the products of the reaction, we may calculate the heat of formation of aluminum borohydride to be -28 kcal/mole. Again, this is not as definite as we would like it to be, and we are continuing with this study to minimize the uncertainties with the hope of generating a more definite value.

B. SAP Study

No studies were performed on this compound during this quarter.

## PROBLEMS ENCOUNTERED

None.

## ACTION REQUIRED BY ONR

None.

## FUTURE PLANS

We are continuing with our hydrolysis studies of aluminum borohydride concentrating our efforts on the low temperature hydrolysis (approximately 1°C), and upon the analysis of the products formed. In addition, we will study the distillation and fractionation process of the sample preparation. Some of our results have led us to suspect this as an area of possible cause of variation of results.

We expect to continue the analytical portion of the SAP study upon receipt of a new sample which is believed to be en route.

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13 ABSTRACT

Several runs of the 2°C hydrolysis of aluminum borohydride with water vapor were performed. The data obtained was more scattered than desirable ($\Delta H$ reaction = -227 ± 7 kcal/mole). We have indications which suggest that the distillation and fractionation temperatures employed in sample preparation may influence this scatter. We are now studying this as a variable. This heat data would yield a value of -28 kcal/mole for the heat of formation of aluminum borohydride.

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